THE ANALYST.

JULY, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of this Society was held on June 7th, at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner occupied the chair.

The minutes of the last meeting were read and confirmed.

It was announced by the Secretary that a letter had been received from the Council of the British Institute of Public Health, cordially inviting Members of the Society of Public Analysts to attend the Annual Congress of the Institute. The Congress would be held this year in Edinburgh, on Thursday, July 27, and the four succeeding days.

The following gentlemen were proposed: As Associates, Mr. Ashworth, assistant to Mr. Smetham, and Mr. Watmough, assistant to Mr. Fairley.

The following gentlemen were duly elected: As Members, Mr. George H. Allibon, Belfast; Mr. Anthony Roche, Dublin; Dr. H. J. Alford, Taunton. As Associates, Mr. C. H. Harrison, Mr. G. E. Scott Smith, Mr. L. K. Boseley.

The following paper was then read by the author:

THE SULPHURIC ACID HYDROLYSIS OF BUTTER-FATS.

By Dr. S. Rideal.

Notwithstanding the various improvements which the Reichert process for determining the volatile fatty acids in butter-fat has undergone, there seems to be room for still further modifications.

Many authors have noticed the ethereal odour which is produced on saponifying the fat with potash in presence of alcohol, and pointed out that a possible loss of volatile acids may take place at this stage of the process. In addition to this possible source of error, the formation of carbonic acid from the alcohol, the presence of acid in the alcohol, and the presence or formation of aldehyde, and consequent production of acetic acid, may tend to raise the number of cubic centimetres of alkali consumed in the final titration. The action of the potash on the liberated glycerine has also been only imperfectly studied, and, finally, the time required to remove the last traces of alcohol further militate against the process. Mr. Richmond (Analyst, 1892, p. 171) has given English readers a good *résumé* of the process and its modifications, but further details on the points just raised will be found treated at length by M. A. J. Zune, in his "Traité Général d'Analyse des Beurres."

The idea of substituting for alcoholic potash sulphuric acid as the hydrolysing

agent seems to have been first worked out by Dr. H. Kreis, of Basel (*Chemiker Zeitung*, 1892, 16, 1,394 ; *Chemisches Centralblatt*, 1893, p. 234), who seems to have argued that the method adopted for the liberation of stearic acid in its ordinary method of manufacture from stearin might be capable of application to butter analysis. He found that butter-fat dissolves in concentrated sulphuric acid at the ordinary temperature, forming glycerine and fatty acids without other products, and that the decomposition takes place much more rapidly than when potash is the hydrolysing agent. The method adopted by him is as follows :

Five grammes of the butter-fat is placed in a half-litre Erlenmeyer flask, and 10 c.c. of concentrated sulphuric acid (sp. g. 1·83) added thereto, and the mixture shaken until clear. The flask is then stood on the water-bath for ten minutes, and 150 c.c. of water added, and the mixture distilled over, using a knee-formed tube without a bulb, but a long 10 cm. stem, into the condenser. The numbers obtained agreed with the Reichert-Meissl method when pure butters were examined, but with margarine and lard higher results were obtained. A genuine butter dissolves clear in the strong acid, but mixtures are usually cloudy at first.

Since the production of the original paper, several foreign investigators have repeated the process, and made modifications in its working.

Since writing this note an abstract of some of these papers has appeared in the last number of THE ANALYST (vol. xviii., p. 165).

Dr. J. Pinette (*Chemiker Zeitung*, 1893, 17, p. 395) found that on repeating the above method a considerable quantity of sulphurous acid was produced, and it naturally tended to give variable and high results. To overcome this difficulty he adds strong permanganate solution before distilling until the pink colour is permanent for a few seconds. The sulphurous acid is then destroyed, and the mixture is distilled as in the Reichert-Meissl process until 110 c.c. have come over.

Pinette and Micko have likewise contributed a paper to the *Zeitschrift für Nahrungs Mitteluntersuchungen* (1893, p. 37), in which they repeat that the Kreis method in its original form gives high results.

Their criticism is also supported by Prager and Stern (*Chemiker Zeitung*, 1893, 27, p. 467), who suggest as a method for removing the sulphurous acid to draw a current of air through the liquid until it ceases to smell of this gas. They quote the following analysis in support of their process :

Reichert-Meissl.		Kreis.		Kreis-Prager-Stern.
1. 30·40	...	41·20	...	31·10
2. 24·81	...	33·50	...	28·76
3. 19·90	...	28·30	...	20·68

These authors remove the sulphurous acid by blowing air through the acid before the addition of the 150 c.c. of water.

To these authors Kreis and Shatzmann have replied (*Chemiker Zeitung*, 1893, 31, p. 544; *vide* also xvii., pp. 395, 468) that the removal of the sulphurous acid is quite unnecessary, provided sulphuric acid of the right strength is employed. Commercial suphuric acid is too strong, and should be diluted until it equals 91·53 per cent.

H_2SO_4. They further add that they have tried Pinette's modification, and find that it gives low results, and quote the following figures :

	Reichert-Meissl.	Kreis-Pinette.
Butter No. 1 ...	... 29·10 ...	18·80
,, ,, 2 ...	... 28·60 ...	17·60
Fused Margarine	... 7·10 ...	5·30

During the last few weeks I have made a series of analyses of butter-fats by the Reichert-Meissl process, and compared the results with that obtained by the use of sulphuric acid, with a view of testing whether the new process is likely to replace it in ordinary work.

The sulphuric acid used had a specific gravity of 1·836, and was free from volatile acids. Our earliest experiments gave high results, which were traced to nitric and nitrous acids present in the sulphuric acid employed. Ten c.c. of acid of this strength were added to 2·5 grammes of melted butter-fat, and gave, on shaking, a clear light-brown solution. In all cases sulphurous acid was produced in sufficient quantity to be recognised by its smell. To this mixture, after waiting a few minutes, 100 c.c. of water, free from carbonic acid, were added and the mixture shaken, when the fatty acids clotted out in white flakes. Permanganate solution was then added from a pipette until the solution attained a pink colour, permanent for a few seconds, the quantity required being seldom more than about 1 c.c. of a strong solution. The distillation was conducted in the flask fitted with a bulb-tube and delivery-tube to condenser bent at an obtuse angle. Eighty c.c. of the distillate were taken for the titration. Pumice-stone, boiled in sulphuric acid, was added to prevent bumping, and on cooling the non-volatile fatty acids solidified to a light-brown cake, which was darker than that of the fatty acids obtained by the ordinary saponification method.

The 2·5 grammes of butter-fat were put into the tared flask when molten, by means of a small pipette with a fine point, and the acid added to the fat.

	Reichert-Meissl.	Sulphuric Acid.
Butter No. 1	... 11·50	... 11·70 and 11·70
,, ,, 2	... 13·30 and 13·70	... 13·70 and 13·70
,, ,, 3	... 13·60 and 13·40	... 14·0 and 14·0
,, ,, 4	.. 15·10	... 14·90 and 15·10
,, ,, 5	... 15·10	... 15·70
,, ,, 10	... 15·60	... 15·70
,, ,, 11	... 14·50	... 14·70
,, ,, 34	... 13·40	... 13·70
,, ,, 35	... 13·80	... 14·0
,, ,, 36	... 13·50	... 13·10
,, ,, 37	... 14·60	... 14·70
,, ,, 38	... 14·90	... 14·10
,, ,, 39	... 12·60	... 12·80
,, ,, 40	... 14·20	... 14·30
,, ,, 25	... 15·70	... 15·90
,, ,, 26	... 15·80	... 15·90
,, ,, 27	... 15·80	... 16·00
,, ,, 28	... 15·00	... 15·00
,, ,, 29	... 15·30	... 14·50
,, ,, 30	... 15·60	... 15·90
Mean 14·42 for Reichert-Meissl, leaving out No 1.		Mean 14·68 for Sulphuric Acid, leaving out No. 1

Here also are a few results which have been obtained with margarines and lards:

	Reichert-Meissl.	Sulphuric Acid.
Margarine No. 1	·80 ...	1·0
,, ,, 2	1·50 ...	1·70
,, ,, 3	1·50 ...	1·60
,, ,, 4	1·10 ...	1·20
Lard No. 1	·30 ...	·40
,, ,, 2	·30 ...	·30 and ·40.

The results for margarine, and also for the genuine butters, seem to point to the new method giving slightly higher results than the standard Reichert-Meissl.

I have to thank my assistant, Mr. Bult, for some of the analytical results which are quoted in this note.

Discussion.

Mr. Alfred H. Allen had been struck with the descriptions of the various modifications of the sulphuric acid process as they appeared in the proof-sheets of The Analyst, and the result was that he instituted one or two experiments upon Pinette's method, in which he used sulphuric acid, and then oxidized the sulphurous acid formed by permanganate. He did not dilute the sulphuric acid, as was now recommended by Schatzmann and Kreis, who reduced it to 1·83 specific gravity, which was almost as great a density as that of the strongest sulphuric acid; but, as had been pointed out, this meant an addition of one-tenth of the volume of water to the strong acid. It was, therefore, necessary to be careful in cases where only the gravity was mentioned. To speak of strong sulphuric acid without specifying the gravity was misleading. His experience, on the few experiments he had made as to the amount of permanganate required, had been very different to Dr. Rideal's. He worked with 5 grammes of butter fat, added 10 c.c. of sulphuric acid diluted, and added a 2 per cent. solution of permanganate, in quantity sufficient to make the mixture pink for a few seconds. The conditions prescribed by Pinette were adhered to, perhaps too rigidly, and consequently a much smaller fraction of the liquid was distilled than in the Reichert-Wollny process. The results did not at all agree with those by the Reichert process, being considerably higher than the latter; but he should certainly try the process again with weaker acid, and under the conditions prescribed by Dr. Rideal.

Mr. Richmond suggested that if, instead of using potassium permanganate, either potassium bichromate or hydrogen peroxide—which were not, as far as fatty acids were concerned, such strong oxidants—were used, the results obtained might come out better. The Reichert process did not satisfy all requirements of the analyst. It was not possible, by means of that process, to detect adulteration with certainty in all cases; and it seemed highly desirable that, instead of attempting to make improvements on this process, analysts should set to work in other directions. There were a good many fields in which he believed information could be obtained which would enable them to get much closer to the detection of adulteration than at present. For instance, he thought there was a great deal to be done by a more detailed examination of the insoluble fatty acids, instead of ascertaining only their

total weight. There were a good many figures which could be obtained on these without much trouble; for example, the saponification equivalent, the iodine absorption, and the freezing-point of fatty acids. The ratio between these would lead to much information. Much might be done in this direction, though it must be remembered that Mr. Hehner had shown that even the separation of the oleic series from the saturated series was by no means easy. Then the solubilities of butter in certain menstrua was useful; in France Bochairy and Honzeau had proposed methods which promised well. The day of single methods was past, and the Reichert process, though it had done good service, could now only be looked on as one link in the chain of evidence.

Mr. Hehner, replying to Mr. Richmond, said that he had every hope of being able before very long to bring the results of a lengthy investigation before the Society, in which he would deal with the suggestions of Mr. Richmond concerning butter-analysis. As regards Dr. Rideal's paper, he might state that some years ago he had also made experiments upon the hydrolysis of butter-fat with sulphuric acid, but could not obtain satisfactory results. It appeared to him that at present the method did not give results which were comparable with those yielded by the Reichert method. In the latter the alkali employed for saponification was neutralized with sulphuric acid, care being taken to have no free sulphuric acid, but only acid sodium sulphate in the liquor to be distilled. On the other hand, by using 10 c.c. of strong sulphuric acid for hydrolysis, none of which was neutralized, it followed that towards the end of the distillation a very strong solution of sulphuric acid was obtained, and consequently there was great risk of sulphurous or some other volatile acid being formed. The sulphuric acid method was inapplicable for estimating the insoluble fatty acids, because the sulpho-oleic acid formed yielded oxy-oleic acid on dilution of the solution, and this latter had a different equivalent from that of the fatty acids obtained by ordinary saponification. He would further point out that, while Dr. Rideal had used 2·5 grammes of fat, with 10 c.c. of acid and 100 c.c. of water, he had the same amount of liquid as was used in the Reichert-Wollny process with only half as much fat, or double as much liquor, as was required for the Reichert process. It was not to be expected, therefore, that the results of the methods could be identical. Dr. Rideal appeared to have, by a happy chance, obtained figures identical with the Reichert figures; but, the conditions being entirely different, the correspondence could be nothing more than a coincidence. He thought that it was exceedingly interesting to have the question investigated from a scientific point of view; but nothing could be much simpler than the method now generally adopted, while the results of the Reichert and Wollny processes were remarkably constant and accurate. It should also not be forgotten that while butter-fat, when mixed with sulphuric acid, evolved but little heat, and only suffered slight decomposition, margarine, which often contained much vegetable fat, would in many cases undergo a much more profound decomposition when acted upon by strong sulphuric acid, accompanied by the evolution of large amounts of sulphurous acid. The process, therefore, was not applicable to anything but pure butter, or to margarine which contained little or no vegetable oil.

Dr. Rideal, in reply, said he did not think there were any questions which he was expected to answer. He quite agreed with Mr. Hehner's remarks as to the quantity of water he had used in his process; but he (Dr. Rideal) wished to use 2½ grammes, and he wished to follow as far as possible Pinette's recommendations, and therefore he had tried in his first experiments the addition of 100 c.c. of water, and then distilling off 80 c.c.; and, finding that the results did agree with the ordinary process, he continued using that quantity. His original idea was, that, as he was dealing with a quantity of strong sulphuric acid, it must be kept dilute. He had not tried any other oxidizing agent. He was glad that Mr. Allen had tried the process, and he hoped that a good many others would do the same.

The Chairman exhibited three photographs of microscopic preparations which had been sent by Messrs. H. B. Stocks and James G. Ross, of 25, Ferndale Road, Liverpool, showing the difference in the crystals of genuine lard, and lard adulterated with beef-stearin obtained by the Barfield method. As the crystals are obtained and examined in ether, the slides cannot be kept for more than a few seconds. Attention was called to the desirability of having a permanent record of these and other samples of food adulteration, since these might be extremely useful in court cases. Messrs. Stocks and Ross expressed their willingness to supply copies of these photographs, or to take photographs of microscopic preparations of any adulterated samples which members might have before them, and of which they wished to keep a record. No price was mentioned.

Papers on the following subjects were also read: "Notes on Rice-Oil and Maize-Oil," "On Rapid Saponification for Analytical Purposes," by Mr. Alfred Smetham; "On Ginger—with special reference to discrimination between genuine and 'exhausted' specimens," by Dr. Bernard Dyer and Mr. J. F. H. Gilbard.

(*Conclusion of the Society's Proceedings.*)

POINTS IN THE ANALYSIS OF CONDENSED MILK.

By H. Droop Richmond and L. K. Boseley.

(*Read at the Meeting, May 3rd*, 1893.)

In an analysis of condensed milk the following determinations are desirable:

Total solids, fat, ash, milk sugar (cane sugar), casein, albumin, total proteids (Ritthausen), and acidity as lactic acid. A determination of total nitrogen is also useful.

The plan we adopt is to weigh out from 15 to 30 grammes of the condensed milk into a 100 c.c. flask, to make up to 100 c.c. and weigh the total; definite weights of the diluted milk will then represent definite weights of the original.

Total Solids and Ash.—Five grammes of the diluted milk are weighed into a basin packed with asbestos, and dried for about 12 to 18 hours (to constancy); the residue is afterwards incinerated (preferably in a muffle).

Fat.—Five grammes are placed on an extracted coil, and the fat estimated by Adams' method; as a check the fat is extracted from the Ritthausen precipitate. The two methods, with care, agree well.

Milk Sugar and Cane Sugar.—These cannot be estimated by the polariscope, as the milk has been heated (ANALYST, xviii. 141). Stokes' and Bodmer's method (ANALYST, x. 62) is suitable, but probably not very reliable (*cf.* Hehner, ANALYST, vi. 218); it is better to weigh the copper oxide reduced before and after inversion with citric acid.

The use of invertase, as proposed by Kjeldahl, would probably be suitable, but we have no experience of this method (*Z. Anal. Chem.*, xxii. 558).

It is a good plan to estimate the reducing powers of a known weight of pure milk or cane sugar, about equal to that in the quantity of condensed milk taken, and to calculate direct from this; in this way all corrections and factors are avoided.

Shenstone's method (ANALYST, xiii. 222) is evidently unreliable, as it involves the use of the polariscope, though in some cases we have obtained satisfactory results with this instrument. We have taken the liberty to correct his sugar figures by dividing the total polarization by 1·042, and they stand then as follows:

	1.	2.	3.	4.	5.	6.	7.
Water	30·3	24·8	26·8	28·0	26·4	25·6	26·8
Fat	4·7	4·7	4·7	4·0	11·5	10·4	10·6
Proteids ...	12·6	12·4	12·4	12·1	12·6	10·7	11·1
Ash	2·1	2·4	2·3	2·4	2·1	1·9	1·9
Milk Sugar ...	15·3	15·7	14·6	14·9	14·4	14·5	14·2
Cane Sugar ...	33·2	36·6	37·8	36·6	28·2	31·8	30·4
Total ...	98·2	96·6	98·6	98·0	95·2	94·9	95·0
Error ...	1·8	3·4	1·4	2·0	4·8	5·1	5·0

We believe it to be quite legitimate to correct the sugar figures thus, as in no part of his paper does Shenstone give even a hint that he departed from Vieth's mode of using the polariscope; one of us (ANALYST, xvii. 222) has already shown this correction to be necessary, and the error introduced by its neglect is large in condensed milk.

Casein and Albumin.—We use Sebelien's method (*Zeit. Physiol. Chem.*, 13, 135)* without modification. The Kjeldahl determinations were made with the addition of a little copper oxide to the sulphuric acid; no sulphide is necessary in the distillation; we find it an advantage to use 30 c.c. of sulphuric acid for the casein precipitate.

* *Sebelien's Method for the Estimation of Casein and Albumin in Milk.*—To 10 grammes of milk add 20 c.c. of a saturated solution of magnesium sulphate and crystals of magnesium sulphate, as long as they dissolve on agitation; a small excess of crystals will not do harm. To obtain correct results, the milk must be as neutral as possible, and the magnesium sulphate free from sodium sulphate (commercial Epsom salts often contain this impurity). The mixture is then allowed to stand for some hours, poured on to a filter, and washed with a saturated solution of magnesium sulphate. The washing can be performed in five to six hours if the filter be not allowed to run dry. The filter, with the precipitated casein, is treated by Kjeldahl's method, using 30 c.c. of sulphuric acid. The nitrogen found, multiplied by 6·37, equals the casein. The filtrate is then diluted, and a few drops of tannin solution (Alumèn's[1]) or phosphotungstic acid solution, added until precipitation is complete. The precipitate is collected on a filter, slightly washed, and treated by Kjeldahl's method. The nitrogen found multiplied by 6·37 gives the albumin.

[1] Alumèn's solution, see ANALYST, xvi., p. 86, footnote.

Ritthausen's Method.—This method is a dangerous one, as the pitfalls are many and not very apparent. It is founded on Ritthausen's observation that the copper oxide compounds of albuminoids are insoluble in water. It is usually directed to use for 10 c.c. of milk 5 c.c. of 6·5 per cent. solution of crystallized copper sulphate, and to nearly neutralize the acid set free; we are of opinion that this quantity of copper solution is excessive, and in the neutralizing a quantity of hydrated oxide or basic compounds are thrown down, and the whole of the matter volatile on ignition is not driven off at 100°—130°, which swells the apparent proteids.

We are inclined to doubt the liberation of *sulphuric* acid, which has a very large heat of neutralization, by an albuminoid; we may mention that Béchamp (*Mémoires prés. à l'Acad. Sciences*, xxviii. 3, 98) is of opinion that casein and albumin exist in milk as salts, and in confirmation of this view that Söldner (*Landw. Versuch Stat.*, 35, 351) has found that in milk there is an alkali in combination as organic salt over and above that required to neutralize Henkel's citric acid (*Molkerei Zeit.*, 2, 259). Milk begins to taste sour and curdles on boiling when the acidity to phenolphthalein is equal to about 0·4 per cent. lactic acid, an amount but slightly greater than that found by Söldner to be necessary. Acting on this view, we have tried neutralizing the milk before adding copper sulphate, with very satisfactory results. We are, however, of opinion that the results thus obtained have a tendency to be a little low, on account of the oxidation of the phosphorus of the proteids to phosphoric acid, which remains combined with the copper on burning. Our Ritthausen results are about equal to the casein and albumin determined separately, and a trifle lower than the total nitrogen multiplied by 6·37. Lately Carcano has published figures showing that Ritthausen's original method is a little higher than the total nitrogen × 6·37 (ANALYST, xvii. 134).

Our procedure is as follows: Dilute 10 grammes of the diluted milk with about 200 c.c. of water, add a few drops of phenolphthalein solution, and neutralize with dilute caustic soda solution, add 2—2·5 c.c. of copper sulphate solution (*as above*), allow to settle, wash about five times by decantation through a tared filter, and then wash on the filter, spreading the precipitate over the filter, dry slightly in the water oven, extract the fat, dry at 130°, burn and subtract the ash.

This modification of Ritthausen's method gives good results with all milk products except whey, which contains albumoses produced by the action of rennet (chymase), which one of us has called chymo-albumoses (*Chem. News*, 67, 132). Neumeister has shown that deutero-albumose obtained during digestion studies by Kühne and Chittenden's method (*Zeit. Biol.*, 23, 381) gives a soluble copper salt, and we have found that the Ritthausen precipitate with whey does not correspond with the total nitrogen multiplied by 6·37. In a mixture of milk and whey in about equal parts we have found about 0·3 per cent. of albumoses precipitated neither by copper sulphate (by our modification) nor by magnesium sulphate, and boiling after slight acidification (to remove albumin); in Sebelien's method albumoses would be estimated as albumin, so that if whey were mixed with milk the Ritthausen precipitate would be less than the casein and albumin, and the albumin would be abnormally high. Chymo-albumoses can be precipitated, however, as very basic copper salts.

Lactic Acid.—We calculate the total acidity as lactic acid.

Of the condensed milks we have analyzed, we may mention one called "The first Swiss brand of unsweetened condensed milk."

This has been analyzed by Faber (*loc. cit.*), and an analysis by a chemist named Goodfellow is given away with the tin.

	R. & B.	Faber.	Goodfellow.
Water	63·47	62·97	61·3
Fat	10·22	10·67	11·7
Sugar	13·94 (diff.)	14·55 (diff.)	14·3
Casein	9·81	9·24	9·1
Soluble Albumin	0·49	0·32	1·5
Ash	2·07	2·25	2·1
	100·00	100·00	100·0

The amount of sugar by polarization was found by us to be 11·5 per cent.

The only important difference in the three analyses is in the soluble albumin, and as great stress is laid upon this large amount in Goodfellow's report, we can only suppose that this chemist is of opinion that his determination represents the amount of soluble albumin in the milk as sold. We can only suppose that he analyzed the milk before sterilization by heat, or that he used some incorrect method of analysis.

Our analysis and Faber's are as concordant as two samples analyzed some years apart could be expected to be. Both the low polarization and the low albumin show plainly that the milk has been heated.

A large class of condensed milks in the market are labelled "skimmed" milk. As these contain "separated" milk, we are of opinion that the label does not comply with the requirements of the Sale of Food and Drugs Act, 1875, section 10. *Skimmed* milk distinctly means that the milk has been set, and the cream that rises has been skimmed off, and this should contain about 1 per cent. of fat.

Separated milk is milk from which the cream has been removed by a separator, and contains about 0·25 per cent. of fat.

The purchaser has a right to expect about 3 per cent. fat in a milk concentrated to one third, as most of these are, and these brands frequently contain much less, though called "skimmed milk."

In our opinion the two words are not synonymous, and are not even so in loose dairy phraseology. It is true that separated milk is often spoken of as "skim" milk, but not to our knowledge as "skimmed" Were these brands labelled as "skim" milk, in our opinion a conviction would be nearly impossible if the use of separated milk were proved; but as they bear distinctly the word "skimmed," which is frequently accompanied by the jesuitical statement that a portion of the fat has been removed, our opinion is that the use of the word "skimmed" might be held to constitute a false label under the Act. It is also possible that the description on the label of many brands, that "a small quantity of cane sugar is added," would also be held to constitute a false label under the Act if the amount of cane sugar exceeded the amount of other solid constituents.

Analyses of such a sample of milk (A) and of a good quality of condensed milk (B) are subjoined:

	A.	B.
Water	29·05	24·86
Fat	1·28	11·28
Proteids	10·63	9·36
Ash	2·33	2·13
Milk Sugar ...	14·91	13·97
Cane Sugar ...	40·07	38·31
	98·27	99·91

Assuming that these milks are concentrated in the proportion 1 : 3, the original composition of the milks was :

	A.	B.
Fat	·43	3·76
Solids not Fat ...	9·29	8·49

The "skimmed" milk is probably badly separated milk, while sample B contains a good proportion of cream.

NOTE ON THE DETECTION OF ADULTERATION OF FRESH MILK BY DILUTED CONDENSED MILK.

By H. Droop Richmond and L. K. Boseley.

(*Read at the Meeting, May 3rd*, 1893.)

Some five years ago a Dairy Farmers' Association offered a gold medal for the solution of this problem. Their chemist advised them that it was not possible, and he had no hope of its ever being done. The medal was not awarded.

Faber (*loc. cit.*) has devised a method of detecting the adulteration by the diminished amount of soluble albumin in heated milk, and we have been able to amply confirm his results.

We have found the albumin to vary from 0·41 to 0·45 per cent. in fresh milks, which is within Faber's limits, 0·35—0·45. The reduction of the rotary power of the milk sugar will also be of great assistance. We would recommend analysts to estimate the milk sugar by the polariscope, and if it falls below 52 per cent. of the solids not fat to make careful gravimetric determinations of the milk sugar by Fehling's solution; to satisfy themselves that the deficiency is not due to lactic fermentation, by acidity estimations; and to estimate the soluble albumin by Sebelien's method.

A discrepancy between the gravimetric and polarimetric estimations of the milk sugar will afford considerable evidence that the milk has undergone heating, and if the soluble albumin is also low the evidence will be almost conclusive.

The limits in fresh milks may be taken as albumin 0·35 per cent., milk sugar and lactic acid 55 per cent. of the solids not fat, and the gravimetric and the polarimetric estimations of milk sugar will not differ more than 0·15 per cent.

The diphenylamine test for nitrates would also afford strong corroborative evidence if impure well water (common on farms) had been used. We operate as follows: Curdle the milk by heating and adding the least possible excess of acetic acid; dissolve a little diphenylamine in *pure* sulphuric acid (1 c.c.), and allow a few

drops of the serum to flow over the surface. A blue colour is characteristic of nitric acid; we have obtained the reaction in a milk to which 5 per cent. of water has been added.

Discussion.

Mr. Faber was very much interested in seeing that Messrs. Richmond and Boseley had taken up the method of analysis which he had some years ago brought before the Society, and which had been proposed by Sebelien. He (Mr. Faber) had only time on that occasion to make a few estimations of albumin in milk—boiled or fresh. He quite agreed with the authors of the paper that the amount of albumin found by Sebelien's method must be absolutely unreliable—that is to say, if the latter meant soluble albumin. He thought, even if Sebelien meant all the albumin present, either precipitated or soluble, the figures were somewhat overstated.

Dr. J. A. Voelcker thought it undesirable to draw a distinction between skim milk and skimmed milk, because it seemed to him that such a distinction would certainly never hold good: not only on account of the similarity of the words, but because all minute descriptions of this kind seemed to him impracticable. As to the test based upon the detection of nitrates and nitrites in the water used, it was quite a frequent occurrence to find water which contained no nitrates at all, and then the test would be useless.

Mr. R. Bodmer, referring to the determination of sugar, wished to know if the citric acid inversion was the only portion of the Stokes and Bodmer process used by them. Many years ago he analyzed a sample of Swiss condensed milk, and had found that it was absolutely free from cane sugar. He would like to know whether the authors of the paper had found the same.

Mr. H. Droop Richmond: Yes, in some cases.

Mr. Otto Hehner expressed the pleasure he felt at seeing Mr. Boseley (a former pupil of his) come forward with a paper. He was glad that the authors had been more successful with the diphenylamine test than he (Mr. Hehner) had been. His difficulty had been that sulphuric acid alone produced an objectionable colour with the milk direct, or with the residue obtained by extracting the milk-solids with alcohol and evaporating the latter. He observed that Mr. Richmond allowed the milk to be tested to flow over the sulphuric acid, avoiding mixture. He noted that the authors drew a distinction between skimmed milk and separated milk. He had learned with surprise that this question had arisen in a court of law, in the prosecution of a vendor of an article labelled "Skimmed Condensed Milk." The sample in question was plainly and honestly labelled in large letters "Skimmed Condensed Milk," the Analyst who gave evidence stating that the contents did not consist of skimmed milk, but of separated milk. The vendor was fined. He (Mr. Hehner) thought that the point was not worthy of the consideration of Public Analysts, who had far more important matters to attend to. The difference between milk that had been hand-skimmed and such that was machine-skimmed was so small that it was not worth talking about in an article like condensed milk, or to make a point of it in a prosecution under the Sale of Food and Drugs Act. Certainly, in most cases, machine-skimmed milk contained a little less fat than hand-skimmed milk, but, on the other hand, separated

milk was quite fresh, while hand-skimmed milk was more or less sour. There were, at the same time, very numerous analyses of skimmed milk on record which showed that often hand-skimming was at least as effective to remove the cream as machine work, and the Analyst could not, therefore, be in a position to prove by determination of the fat alone what method of skimming had been employed, unless, indeed, the whole of the fat were removed, which was not the case in the sample subject to prosecution. No two separators worked exactly alike, and even the same machine gave different results on different days. Therefore, if the Analyst concerned carried out his contention to the logical extent, he would have to insist upon the name of the machine used being stated, as well as the number of revolutions of the separator. His decided opinion was that, due notice having been given by a plain and honest label that the fat had been removed by skimming, the purchaser was not prejudiced, no matter whether the skimming had been done by hand or by separator, or more or less completely. The purchaser had no right to expect in skim-milk any fat at all, and any that was left in the skim-milk was really a present to him and a loss to the manufacturer of the condensed milk. No ordinary purchaser expected any fat in skim-milk at all. He thought the point was a mere quibble, and not exactly fair to the merchant who tried to conduct his business honestly. At the present time there was no consensus of opinion, even among experts, in this matter, and unless by common consent a distinction was made between skimmed and separated milk, it was not right to spring such a point in a court of law upon the vendor. He was strongly opposed to the sale of skimmed condensed milk, unless due notice was given to the buyer, but such notice having been given plainly and unmistakably, it appeared to him quite immaterial how and to what extent the skimming had been effected. The absurdity of the position was made plain by the attempt of the authors of the paper to make a distinction between skimmed milk and skim-milk.

Mr. Cassal disagreed with Mr. Hehner. He could not admit that because Public Analysts had a number of other things to deal with, therefore this matter should be left alone. The Public Analyst had to deal with the things which were submitted to him. It happened that certain public Authorities were in the habit of deciding what articles should be analyzed, and it was not advisable for a Public Analyst to object to that course of action. The persons responsible for the taking of samples in such cases as those referred to were the Committee of the Authority. It invariably threw suspicion on a public officer if he raised objections to any course which was desired by a Committee or a Board. The case referred to was only one among many. These articles were generally sold under the name of "Condensed Milk," and in some corner of the label, printed in small letters, there was a statement containing the word "skimmed," the word being introduced on some other part of the label than that containing the principal title. Statements of that sort no one could possibly defend. If a condensed milk was practically devoid of fat, that fact should be stated fully on the label, and should form part of the title of the article. The use of the word "skimmed" was not a sufficient disclosure of the real nature of such an article, and the label that Mr. Hehner had alluded to did not contain a sufficient disclosure. Why was it that a certain class of vendors desired to adhere to the use of the word "skimmed" and would not use the word "separated," which

accurately disclosed the nature of the article? The only answer was that the word "skimmed" conveyed a more favourable impression, and that, in fact, the article so described would be thought better than it really was. Did anyone suppose for a moment that the manufacturers of these articles employed an army of persons to hand skim their milk before they condensed them? It was a matter of common knowledge that these articles were produced by means of separators, and that they could only be so produced. When Mr. Hehner said that there was but very little difference between the percentages of fat found in milk that had had cream removed by hand and in milk treated by a separator, he forgot also to point out that it was possible to get any results one liked with a separator. It depended on how far it was worked; and the comparison which he made between the percentages of fat were entirely beside the mark, because, in the first place, the figures referred to had been collected from all sources—they were not authenticated, and there was no doubt that many had been obtained by obsolete processes and unreliable persons; and, in the second place, the matter in hand was the comparison between legitimately hand-skimmed milk and milk which had been almost completely deprived of fat, a result which had unquestionably been attained by means of a separating machine.

Mr. Allen quite agreed that the point which had been raised was one of very great interest to the Society of Public Analysts; and the opposite views taken by Mr. Hehner and Mr. Cassal showed how much need there was for discussion. A point which had not been referred to was the misleading statement to be found on the labels of many brands of condensed milk respecting the amount of water required to be added. This was often very greatly overstated, with the consequence that the consumer had milk of far less nourishing power than he supposed.

Mr. E. J. Bevan said that he did not agree with Mr. Hehner, and not altogether with Mr. Cassal. It might be true, as Mr. Hehner had said, that a large number of so-called separated milks certainly showed a higher percentage of fat than skimmed milks. Everything depended upon the way in which one operated. But taking the average composition of separated milks and the average composition of skimmed milks, there was not a shadow of a doubt that the proportion of fat removed in the case of the separated milk was much greater than in the other case. This went a great way towards showing that there was a definite distinction between the two. Some of the samples labelled "Condensed Skimmed Milk" which he had received contained a very small proportion of fat, so small that he could have no doubt that they had been treated in a separator.

Dr. Voelcker hoped that Public Analysts would not attempt to draw distinctions between so-called skimmed and separated milks. He regarded skimmed or separated milk as milk from which the fat had been removed, and in his opinion it was a matter of accident as to how much had been removed. If it could be said that milk always contained a definite quantity of fat when skimmed by hand, and another definite quantity when separated by machine, then clear distinctions could be drawn, but not otherwise.

Mr. Faber said that separated milk contained an extremely low amount of fat, which would not exceed from ·1 to ·3 per cent. in well-managed dairies. If milk had

to be hand-skimmed before it commenced turning sour, so as to be fit for condensing, it would be found that the skimmed milk contained sometimes as much as 2 per cent. of fat, very often over 1 per cent. He considered that there was a great deal of difference between hand-skimmed milk and separated milk. There was a point in favour of separated milk which might be mentioned, and that was that by separating, not only was the fat removed, but the impurities also to a very large extent. On the inside of the separator bowl an aggregation of very dirty slimy black matter was formed, which contained almost all the dirt present in the milk, and which was thus kept out of separated milk. The value of skimmed milk, as a nourishment, varied to a certain extent with the contents of fat. With regard to the label, if it merely stated that a portion of the fat had been removed, and the amount removed was found to be something like 95 per cent., then he considered it a misleading label, but if the contents of the tins were described on the label as "Skimmed Milk," then he thought this was a sufficient disclosure.

Mr. Cassal thought that Dr. Voelcker's remarks were much to the point, as they admitted the accuracy of his (Mr. Cassal's) contention. Dr. Voelcker had said that the amount of fat left by hand-skimming was uncertain. He had also said that the amount of fat which might be left in milk after using a separator was uncertain. But there was one thing which was certain, and which he understood that Dr. Voelcker and Mr. Hehner admitted without reserve, and that was that when there was practically no fat at all, when 90 per cent. of it had been abstracted, then there was no doubt whatever that the abstraction had been effected by a separator. This was all that he (Mr. Cassal) required. Mr. Faber had published in THE ANALYST some years ago a paper in which he advocated the use of the term "separated" for these articles, and in which he said that the word "skimmed" did not afford a sufficient description of them.

Mr. Faber admitted that he had in the paper referred to said that the term "condensed separated milk" would be a more correct description than if it was merely mentioned that "part of the cream" had been abstracted, but he had not used the word "separated" as against the word "skimmed." The difference was between printing on the label in large letters "Skimmed Milk" or "Separated Milk," and of calling the milk with such a name as, for instance, "Goat Brand," and then in small letters adding that "part of the cream" has been abstracted.

Mr. Cassal gathered, from what Mr. Faber had just said, that he adhered to his original recommendation, and that he thought it would be better for the public if these articles were described by their correct name, as "Condensed Separated Milk."

Mr. Allen said he had been struck with the ingenious and powerful manner in which the arguments had been put on both sides. With reference to the whole subject, he would point out that the Government had promised a Select Committee to consider Dr. Cameron's Bill now before the House of Commons as soon as it had passed its second reading. In view of the fact that the Government appeared to be desirous of dealing with the subject of adulteration, although they could not do it very thoroughly on the basis of such a Bill as Dr. Cameron's, it was important that Analysts should be agreed as to how articles should be labelled. He was

afraid that there were not very many people among the lower classes who knew the meaning of the words "separated" milk, and, for his own part, he preferred the term "skimmed" milk. He thought "skimmed milk" covered the whole ground and *included* separated milk, which might be regarded as mechanically skimmed milk. There might be less fat in the mechanically than in the hand skimmed milk, but it would be difficult to establish a definite distinction between them which would stand critical examination. He thought the distinction attempted to be drawn between the words "skim" and "skimmed" was a quibble. They ought to base their action on much broader grounds than these, which were objections only worthy of lawyers. With regard to the estimation of nitrogen in the copper precipitate obtained by Ritthausen's method, he thought that if the precipitate—including the filter-paper, if they liked—were treated by Kjeldahl's process, a much more reliable result would be obtained than by igniting the precipitate and regarding the loss as proteids.

Mr. Richmond, in reply, said that, with regard to Mr. Allen's suggestion of estimating the nitrogen in the Ritthausen precipitate, he had sometimes done so; it seemed to be a more satisfactory method of estimating proteids by actual weighing than by calculation from the nitrogen; the percentage of nitrogen in casein was not known with absolute accuracy, Hammarsten giving 15·65 per cent. and Chittenden and Painter 15·9 per cent., and the error of the factor would be probably as great as the error of weighing. A remark had been made to the effect that in the adulteration of fresh milk by condensed skimmed milk it was not reliable to depend on the presence of nitrates. In their paper they had brought this forward as affording further and independent evidence of adulteration; if nitrates were found it was very strong, and, in fact, the only direct, evidence that the milk had been mixed with water (containing nitrates). Mr. Hehner had asked if they had examined samples of milk which had been purposely adulterated with diluted condensed milk. They had not done so. In a diluted condensed milk they had found a deficiency of 0·8 per cent. of sugar. This paper was not intended as an exhaustive one, it was only called a "note," and they thought that the evidence would induce others to make experiments. The chief part of the discussion had turned upon what they (the authors of the papers) considered a somewhat unimportant point—whether "skimmed" and "separated" milk were synonymous terms. Mr. Boseley and he held no very strong opinion on this point, and had introduced it for the sake of eliciting the opinion of the Society; the subject had some practical interest, as cases had lately occurred. They had stated subsequently that if the words "Skim Milk" were used on the tins, it might be difficult to obtain a conviction, but between "skimmed" and "separated" milk there was a difference. He observed the point was not seen by some. There were two distinct things—skimmed milk and separated milk. Two statements to the contrary had been brought before the meeting, *i.e.*, König's analysis and Dr. Voelcker's experiments at shows. He (Mr. Richmond) did not think that the results given by a machine fitted up at a show could be taken as a fair specimen of what the machine could do, as it was worked frequently under unsuitable conditions, and by operators whose minds would be occupied by details unconnected with the working of the machine. König's

figures were extracted from all kinds of sources, and the majority of the determinations made by methods now admitted to be inexact. With regard to the extremely low percentage of fat sometimes found in hand-skimmed milk, that low percentage could be obtained if desired, but it could not be done on a commercial scale. If one considers that the milk in the tins is always fresh, he thought there could be no shadow of doubt that skim milk would contain more fat than separated milk. Personally, he could echo a remark of Mr. Hehner's, that it was not a serious infringement of the Act, and that there were other adulterations which were more in need of being stopped.

VINEGAR.*

By Alfred H. Allen, F.I.C., and C. G. Moor, B.A., F.C.S.

Recent prosecutions for the sale, as vinegar, of an article consisting essentially of acetic acid obtained by the distillation of wood, have caused pharmacists generally to consider their position with regard to such an article, and to inquire what is properly to be understood by the term "vinegar."

Ordinary dictionary definitions of technical products are not unfrequently unreliable, but it is nevertheless of interest to note the description of vinegar given in some of our leading dictionaries and encyclopædias.

The "Imperial Dictionary" gives the following definition and description of vinegar:

"Dilute and impure acetic acid, obtained by the vinous fermentation. In wine countries it is obtained by the acetous fermentation of inferior wines, but in this country it is usually procured from an infusion of malt which has previously undergone the vinous fermentation. Vinegar may also be obtained from strong beer, by the fermentation of various fruits, or of a solution of sugar mixed with yeast; in short, all liquids which are capable of the vinous fermentation may be made to produce vinegar."

The "Encyclopædia Britannica" states that—

"Vinegar is a dilute form of acetic acid, having a flavour that varies according to the source from which it is obtained. . . . The nature of acetous fermentation, and the *rationale* of the processes by which vinegar is prepared, are explained under 'Fermentation'; and the acetic acid obtained by the destructive distillation of wood is dealt with under 'Tar.' Here we have only to deal with the various kinds of vinegar used for table, medicinal, and other household purposes."

In the first edition of Watts's "Dictionary of Chemistry" vinegar is classed under the separate heads of "Wine Vinegar," "Malt Vinegar," "Distilled Vinegar," and "Wood Vinegar." In the new edition there is no distinct definition of vinegar. Under the preparation of acetic acid is the following passage:

"Under the influence of ferments.—This is the ordinary process of making vinegar from alcoholic liquids, wine being generally used for the purpose in France and Germany, and malt in England."

* Abstract of a paper in the *Chemist and Druggist*.

T. E. Thorpe, in his "Dictionary of Applied Chemistry," vol. i., states that—

"In all processes for the manufacture of vinegar advantage is taken of the oxidizing action of the vinegar-fungus already described; the souring of wines and other alcoholic liquids is due to this organism, the germs of which are always present in the air, and are deposited, and grow in any suitable medium."

A. H. Allen, in his "Commercial Organic Analysis," vol. i., states that—

"Properly speaking, vinegar is a more or less coloured liquid, consisting essentially of impure dilute acetic acid, obtained by the oxidation of wine, beer, cider, or other alcoholic liquid. Sometimes the term is improperly extended to pyroligneous acid or 'wood vinegar,' while acetic acid is called 'distilled vinegar.'"

S. P. Sadtler, in his "Industrial Organic Chemistry," p. 231, says:

"Only such materials will be considered here as give rise to a vinegar by the normal acetic fermentation. The manufacture of acetic acid and technically important acetates will be spoken of later, under pyroligneous acid, as derived from the destructive distillation of wood. The materials referred to as furnishing vinegar under the influence of the acetic fermentation are—first, wine; second spirits; third, maltwort or beer; fourth, fermented fruit-juices other than wine; and, fifth, sugar-beets."

W. T. Brannt, on page 20 of his "Practical Treatise on the Manufacture of Vinegar and Acetates," says:

"For consumption on a large scale, especially where only a body of an acid taste is required, the use of so-called 'vinegar essence' (*i.e.*, pure 80 or 90 per cent. acetic acid), prepared from wood, and which, when properly diluted, furnishes ordinary vinegar, will undoubtedly gradually supersede vinegar prepared from alcohol, it being considerably cheaper. And, notwithstanding that the price of vinegar essence is decreasing every year, in regions where wood is plentiful and cheap its manufacture is a well-paying industry, on account of the many valuable by-products (tar, wood-spirit, charcoal) obtained besides acetic acid. Even at the present time for all industrial purposes where acetic acid is required—as, for instance, in the manufacture of tar colours—that obtained from wood is used, and the quantities consumed in the fabrication of table vinegar become larger every year. But the manufacture of vinegar from alcohol and alcoholic fluids will nevertheless continue to flourish, because the product obtained from them actually possesses different properties from the pure acetic acid prepared from wood. Vinegar obtained from pure alcohol, and still more so that from fermented fruit-juices, as wine, cider, skins of pressed grapes, or from malt, contains, besides acetic acid and water, small quantities of bodies which, on account of their being analogous to those occurring in wine, may be designated as 'bouquet-bodies,' and which give to the vinegar an agreeable smell and taste, entirely wanting in acetic acid prepared from wood. These properties are so characteristic that anyone gifted with a sensitive and practised sense of smell can at once distinguish pure acetic-acid vinegar from that prepared from wine, cider, beer, etc. By the addition of volatile oils or compound ethers an agreeable odour can, of course, be imparted to vinegar obtained by diluting pure wood acetic acid with water, but it is impossible to produce the harmonious bouquet peculiar to vinegar from alcohol or fruit-juices, a similar relation existing here as between wine and so-called artificial wine. The latter can be made so as nearly to approach, as regards taste and smell, genuine wine, but a connoisseur will at once detect the difference."

In "Chemistry applied to the Arts and Manufactures," edited by Charles Vincent, wine vinegar, malt vinegar, fruit vinegar, and pyroligneous acid or wood vinegar, are described in separate articles.

In Spon's "Encyclopædia of the Industrial Arts, Manufactures, and Commercial Products," page 2038, the following passage occurs:

"Vinegar is an acid liquid, described in the British Pharmacopœia as prepared from malt and unmalted grain by acetous fermentation. The acid contained in vinegar is acetic acid, and it usually exists in the proportion of 3 to 6 per cent. . . . Although the official prescription is adhered to by some manufacturers, the use alone of those ingredients is by no means usual; indeed, malt, in many instances, is not in the present day used at all, but for it are substituted artificial glucose and cane-sugar or molasses. These latter are very largely used, and as they produce, chemically speaking, the same result—*i.e.*, acetic acid obtained by fermentation—there can really be no objection to their use."

The "National Dispensatory" of Stillé and Maisch states that—

"Vinegar is a dilute acetic acid, obtained by the acetic fermentation of alcoholic liquids. The materials from which it is prepared influence its colour, and to a considerable extent also its odour and taste."

In Pereira's "Materia Medica" it is stated that—

"The acetic acid of commerce is derived from two sources—vinegar and pyroligneous acid; the first is procured by exciting the acetous fermentation in certain liquids, the other by the distillation of wood."

A. Wynter Blyth, in "Foods: their Composition and Analysis," describes under "Varieties of Vinegar" the various fermentation-products, and classes pyroligneous acid among the adulterants of vinegar.

A. H. Hassall, in his "Food; its Adulterations and the Methods for their Detection," published in 1876, gives the following "definition of adulteration" of vinegar:

"Free sulphuric acid beyond the quantity allowed by law, or any other mineral acid, or vegetable acid, excepting acetic acid, derived from the manufacture of the vinegar; water in such proportion as to reduce the acetic acid to below 3·5 per cent. It is questionable whether it would not be well that the law should be abolished which allows of the addition of one part of sulphuric acid in 1,000 parts of vinegar."*

On page 629 of the same work Hassall states:

"The different kinds of vinegar may, according to their source, be divided and classified as malt, wine, cider, beet, sugar, and wood vinegars; but occasionally other fruits, as gooseberries and currants, are employed for the manufacture of vinegar."

From this passage, taken alone, it might conceivably be held that Hassall regarded acetic acid from wood as a true variety of vinegar, but the definition already given excludes this interpretation, in addition to which Hassall states, a few pages later (635), that—

"The principal adulterations of vinegar are with water, sulphuric acid, burnt sugar, and sometimes with acrid substances, as chillies and grains of paradise, and also with acetic and pyroligneous acids."

On the following page (636) he enters into greater detail, thus:

"Very commonly, after the manufacture of vinegar has been completed, the strength is brought up by an addition of acetic acid. We are of opinion that this

* This statement respecting the legality of adding sulphuric acid to vinegar is of very doubtful accuracy. So far as I can ascertain the fact, the addition of 1 gallon of sulphuric acid to 1,000 gallons of vinegar was formerly permitted under an Excise regulation, but the addition being found unnecessary for its intended purpose, the permission was subsequently withdrawn. One gallon per 1,000 was of course very different from 0·1 per cent., the quantity commonly said to have been allowed.

practice is to be regarded as an adulteration. To allow of this addition would be to acknowledge that a mixture of acetic acid and water really constitutes vinegar, which is far from being the case, since genuine vinegar contains extractive matters of different kinds, as well as certain volatile principles, and which (*sic*) affect both the aroma and the flavour."

In the British Pharmacopœia vinegar is defined as "an acid liquid, prepared from a mixture of malted and unmalted grain by the acetous fermentation." The specific gravity is from 1·017 to 1·019, and it is to contain about 5·41 of real acetic acid ($C_2H_4O_2$).

The vinegar of the German Pharmacopœia is required to contain at least 6 per cent. of absolute acetic acid. In Russia the minimum limit of strength is 5 per cent.; in Austria, 6; in Belgium, 5·6; in France, 8 to 9; and in the United States, 4·6 per cent.

In 1874 the Society of Public Analysts adopted 3 per cent. of real acetic acid as the minimum limit of strength for vinegar.* This limit certainly cannot be said to err on the side of too great stringency, and there have been very few prosecutions for the sale of vinegar containing less than this very moderate proportion of acetic acid.

With regard to the sale of vinegar, the pharmacist stands in a far more delicate position than the general dealer, for it might be argued, with some plausibility, that, when purchased of a registered pharmacist, an article recognised in the British Pharmacopœia ought to comply with the description of it given by that authority. This would limit the "vinegar" to be sold by pharmacists to the very best quality of malt vinegar; and if the B.P. definition of vinegar were legally applied to-day, a considerable proportion of the trade would probably be caught tripping.

With the single exception of Brannt, who appears to hold that vinegar may be legitimately manufactured from wood acid, while admitting that the product is inferior to the fermentation acid, all the authorities above quoted agree in regarding true vinegar as an acid liquid produced by the acetous fermentation of alcoholic liquids, and consequently regard acetic acid from wood as not answering to the description of genuine vinegar.

Detection of Cotton-seed-Oil in Lard. F. Gantter. (*Zeitsch. f. anal. Chem.*, xxxii. 303.)—The author states that he has met with several samples of cotton-seed-oil which failed to give a silver-reaction, and also a number of samples of lard strongly suspected of adulteration with cotton-oil, which gave no indications by Becchi's test. He concludes that some treatment of the oil is now adopted with a view to circumvent analysts. He has modified the sulphuric acid test, which depends upon the dark coloration produced with cotton-oil, but not with lard, and has in this respect followed Flückiger (*Zeitsch. f. anal. Chem.*, vol. x., p. 235), who recommended dilution of the oil to be tested with ether, benzol, chloroform, or carbon bisulphide, in order to moderate the action of the sulphuric acid. He takes 1 c.c.

* By the term "real acetic acid" there is good reason to believe that acetic anhydride was intended. Three per cent. of $(C_2H_3O)_2O$ corresponds to 3·53 per cent. of $(C_2H_3O)OH$.—A. H. A.

of the oil or fat to be tested, adds 10 c.c. of petroleum ether, and a single drop of strong sulphuric acid, shaking violently at once. Pure lard becomes straw-coloured, or faint reddish-yellow; after some time the solution becomes clear and colourless, or nearly so, while dark-red droplets separate. Cotton-oil at once becomes dark-brown or black, and the solution remains so for a very long time. Mixtures take on a more or less dark-brown shade; even 1 per cent. of cotton-oil mixed with lard showing the reaction plainly. Olive-oil behaves in a similar manner to lard, but earth-nut-oil resembles cotton-oil.

The author also gives a number of iodine figures for pure and mixed lard, which differ considerably from those found by others. He is of opinion that pure lard should not absorb more than 27 per cent. of iodine. O. H.

Note by the Abstractor.—The coloration produced by sulphuric acid is almost entirely dependent upon the quantity of sulphuric acid added. A sample of lard, dissolved in petroleum ether, as directed by the author, remained uncoloured by a single small drop of sulphuric acid, with a large drop became yellowish, and brown with two drops. The same changes were observed with a number of other samples. It appears to the abstractor that neither the method nor the iodine figures given by the author are trustworthy.

The Detection of Saccharin in Beer. F. Gantter. (*Zeitsch. f. anal. Chem.*, xxxii. 309.)—Börnstein (*Zeitsch. f. anal. Chem.*, vol. xxvii., p. 167) has recommended, as a most delicate reaction for saccharin, heating the substance to be tested with resorcin and strong sulphuric acid, fluoresceïn being formed. The author, who examined a number of samples of beer for saccharin, found that the reaction is not conclusive of the presence of saccharin, since beer perfectly free from that body yields a strong fluorescence when the residue from the acid and ether treatment is heated with resorcin and sulphuric acid. He finds that hop-resin and ordinary colophony behave similarly in this respect to saccharin. He relies solely upon the taste of the residue, and operates as follows: 500 c.c. of beer are evaporated to a syrup, precipitated with alcohol, the alcoholic solution acidified with HCl, and then evaporated. The residue is shaken out with ether, the solvent evaporated, and the residue tasted. He is of opinion that the method proposed, which depends upon the detection of sulphur in such residue, is of no use, since the amount of saccharin which is likely to be present in half a litre of beer will be exceedingly minute; consequently but faint indications of sulphuric acid will be obtained after fusion with carbonate and nitrate of potassium. O. H.

On a Method of Titrating Boric Acid. R. T. Thomson. (*Journ. Soc. Chem. Ind.*, xii. 432.) -It is well known that boric acid, when tested in aqueous solution, is neutral to methylorange and slightly acid to phenolphthalein, but as the end reaction is very indistinct, it has not hitherto been found possible to titrate the amount of boric acid alkalimetrically. The author finds that when glycerin is added to a solution of a borate the acidity is increased until, when about 30 per cent. of glycerin, calculated upon the total amount of fluid, is present, the maximum

acidity is reached and the total amount of boric acid is sharply indicated, phenolphthalein being the indicator. The compound $NaBO_2$ is produced; 1 c.c. of standard soda solution, therefore, corresponds to 0·0620 grammes H_3BO_3.

When boric acid is to be estimated in borax or in mixtures of borax and boric acid, an aqueous solution is made, methylorange added, and standard acid to neutrality. Thus the total amount of combined alkali is obtained. The whole of the boric acid is now free. Phenolphthalein is now added, and sufficient pure glycerin until the amount of the latter is at least 30 per cent. of the total fluid, and the acidity titrated, 1 c.c. being equal to 0·0505 $Na_2B_4O_7$, or 0·0955 $Na_2B_4O_7 + 10H_2O$. In the case of the boric acid of commerce, which generally contains salts of ammonia, 1 gramme may be dissolved in hot water, a slight excess of sodium carbonate added, and the solution boiled down to about half its bulk to expel all the ammonia. Any precipitate which forms is filtered off and the filtrate titrated as described.

Boracite or borate of lime is first dissolved in dilute hydrochloric acid, the solution nearly neutralized with caustic soda, the solution boiled to expel carbonic acid, cooled, exactly neutralized, using methylorange, and the boric acid titrated as directed. If much iron be present, this may be removed by a preliminary treatment with carbonate of sodium, and the oxide of iron, as well as carbonates of calcium and magnesium, precipitated and filtered off.

The author is making experiments as to the applicability of the method for the determination of boric acid in milk and other food materials. This new method appears to furnish a satisfactory solution of an important and difficult problem.

O. H.

REVIEW.

Traité Pratique d'Analyse Chimique et de Recherches Toxicologiques, par G. Guérin. London: Ballière, Tindall and Cox.

This compendious work covers 470 pages, exclusive of the index, and contains much information on qualitative analysis not usually found within the same covers. The book is typical of the faults and merits of French works on chemistry. Formulæ and equations are conspicuous by their absence throughout the greater part of the work, and in many cases where they might have been employed to great advantage, but graphic formulæ are given of pilocarpine and certain other of the alkaloids. Alkaloids, by the way, are grouped together with the glucosides and other non-basic bitter principles, without a clear distinction being drawn between them. The ptomaines and leucomaines are duly described.

In the portion of the work devoted to inorganic compounds, the ordinary reactions of the metals and acids are fully described, and the rarer metals are afforded greater prominence than is usual in English text-books. The almost entire absence of formulæ in this part of the work leaves the student in ignorance of the exact composition of the precipitates he produces, and seriously detracts from the value of the book. The information given is often tantalizingly incomplete, and occasionally incorrect. Thus, on page 59, the reader is correctly told that lead chromate is soluble in potash, but insoluble in acetic acid, and that barium chromate is soluble in hydro-

chloric and nitric acids; but the further valuable information that the former precipitate is soluble in nitric acid and the latter insoluble in acetic acid is not given. On page 61 the characteristic reactions of cuprous salts with iodides and thiocyanates are not referred to. Schlagdenhauffen's reaction of magnesium salts with sodium hypoiodite (prepared by dissolving, immediately before use, powdered iodine in a 2 per cent. solution of caustic soda until the liquid is coloured strongly yellow) is not well known in England. The reagent gives a voluminous brown-red precipitate with magnesium, or in a very dilute solution a reddish coloration, while lime, baryta and strontia are unaffected. The most important practical reaction of titanium, namely, that of forming a solution in sulphuric acid, or acid sulphates of alkali metals, from which metatitanic acid is precipitated on dilution and prolonged boiling, is entirely omitted. Under zinc, no reference is made to the delicate reaction of hot ammoniacal solutions with potassium ferrocyanide. In describing the reaction of acetates with ferric chloride, the red liquid is said to yield a brown precipitate of ferric hydrate on boiling. No attention is given to the quantitative possibilities of the various reactions described, and the methods for separating the metals and salt radicals are dismissed very summarily, as are the means of dealing with insoluble substances. Methods for qualitatively analyzing gases are given, and there is a chapter on spectrum-analysis, in which the micro-spectroscope is not described. This is followed by methods of toxicological analysis, in which the usual processes for detecting arsenic, phosphorus, hydrocyanic acid, chloral and chloroform are detailed. The isolation of vegetable alkaloids is next described, and the inevitable tables of Dragendorff reproduced. The work closes with a description of "methods and practical processes for the analysis of some substances necessitating more special treatment." The substances considered in this section are limited to water, clay, steel and iron. On page 402 we are told that in 1847, in England, M. Clarck (*sic*) indicated a method of rapidly examining waters, and that this method has been perfected in France by MM. Boutron and Boudet, who have written of it under the name of "hydrotimetry." This is simply Clark's soap test, the only apparent innovation being the use of the instrument long obsolete in English laboratories, formerly known as a Binks' burette. A full description is given of the methods of effecting a bacteriological analysis of water.

M. Guérin's work appears to be very unequally written. The whole of the information relating to the commoner metals and acids is given more fully and accurately by Fresenius, but in the case of the less frequently occurring metals and salt-radicals M. Guérin has recorded much useful information. Of quantitative processes he seems to have a wholesome horror, and even when, as in the determination of the saline matters in water, he is compelled to weigh such compounds as barium sulphate, magnesium pyrophosphate and potassium chloroplatinate, he gives no formulæ for these products, and carefully abstains from indicating the method of deducing from the weights obtained the corresponding amounts of the substances sought to be determined.

The book contains a good deal of useful information, and the faults and omissions which detract from its value as a handbook for students, for whom it is professedly written, will not be felt by the skilled chemist, who, on the other hand, will learn how little the work of English analysts is known or appreciated in France. Many of the illustrations will be welcomed as old friends. Excellent coloured diagrams of borax beads and metallic spectra are given.

A. H. A.

CORRESPONDENCE.

To the Editors of THE ANALYST.

SIRS,—I send you the following, in the hope that it may be of interest to your readers, and possibly of service if any of them should be similarly situated.

On the 5th day of May, 1879, I was appointed Public Analyst for the Borough of Neath, as per following resolution, a copy of which was forwarded to me by the late Town Clerk :

"Resolved, that Dr. William Morgan, Public Analyst of Swansea, be appointed Public Analyst for this borough, on the terms mentioned in his letter of the 16th April ult."

The terms were 10 guineas per annum and 15s. per sample. In due course the sanction of the Local Government Board was given, as provided under section 10 of the Food and Drugs Act, 1875.

All went on well until December, 1892, when a resolution was passed, at a meeting of the Neath Town Council :

"That Dr. Morgan be given three months' notice, as from the next quarterly meeting of this Council, to terminate his engagement as Public Analyst for this Borough."

The purport of the above was to terminate my appointment in May, 1893.

The above resolution was forwarded to me by the Town Clerk, to whom I replied as follows :

"I have duly received your letter containing copy of resolution of the Town Council of the borough of Neath, purporting to terminate my appointment as Public Analyst three months from the first Monday in February next. I must, however, respectfully decline to accept such notice until my removal has been sanctioned by the Local Government Board."

The above letter was read at the January Council meeting, and the following resolution was passed :

"That the Town Clerk do inform the Local Government Board that the appointment of Dr. Morgan as Public Analyst for this borough has been cancelled."

The following is the reply of the Local Government Board :

"I am directed to acknowledge the receipt of your letter of the 3rd inst., embodying a copy of resolution passed by the Town Council respecting their proposal to cancel the appointment of Dr. Morgan as Analyst. In reply, I am directed to point out that the appointment of an Analyst, under section 10 of the Sale of Food and Drugs Act, 1875, cannot be determined by a Local Authority without the approval of the Board. Before further considering the subject, the Board request to be fully informed of the reasons of the Town Council for desiring to dispense with Dr. Morgan's services."

At a Council meeting held on the 6th day of February, 1893, the foregoing letter was read, and it was resolved to reply to it as follows :

"1st. We have an Analyst who can be seen at any time by the officers of the Corporation.

"2nd. There is a greater possibility of the townspeople taking advantage of the services of an Analyst who lives in the town.

"3rd. That as local autonomy is the order of the day, the Corporation are justified in terminating the engagement of their officers at pleasure, and are certainly entitled to prefer the employment of a townsman to that of a stranger."

The following reply, dated March 6th, was sent by the Local Government Board to the Town Clerk :

"I am directed by the Local Government Board to acknowledge the receipt of your letter of the 9th ult., and of its enclosure, with reference to the proposal of the Town Council of the borough of Neath to determine the appointment of Dr. Morgan as Analyst for the borough.

"In reply, the Board direct me to state that there do not appear to them to be any sufficient grounds to justify them in approving of Dr. Morgan's removal from office, and that they are therefore unable to accede to the application of the Town Council."

The above letter was read at the Council meeting held on the 12th April last, and it was resolved to send the following to the Local Government Board :

"That the Local Government Board be informed that Dr. Morgan was appointed under section 11 of the Food and Drugs Act, 1875, and not under section 10; that no confirmation of his appointment was necessary; and that the information conveyed to them as to determining the appointment was merely an act of courtesy on the part of the Town Council."

The following is the reply of the Local Government Board, and read at the meeting held May 1st :

"I am directed to state that the Board have referred to the correspondence, and to the copy of the resolution passed by the Town Council with regard to the appointment of Dr. Morgan, and that they see no ground for the contention that the appointment was not made under section 10 of the Sale of Food and Drugs Act, 1875."

The last resolution of the Council is, "That a copy of the whole of the correspondence be sent to one of the County Members, in order that he may question the President of the Local Government Board in the House of Commons."

I may state that the sole object of the Council was to remove me in order to give the appointment to a young man residing at Neath, and whose father and brother are aldermen of the borough.

I am, yours faithfully,

WILLIAM MORGAN, Ph.D.

Swansea, May 3rd, 1893.